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मानक

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IS 517 (1986): Methanol (Methyl Alcohol) [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 517 - 1986
(Reaffirmed 1996)

Indian Standard
SPECIFICATION FOR
METHANOL (METHYL ALCOHOL)
(Second Revision)

Third Reprint JANUARY 2002

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

**AMENDMENT NO. 1 NOVEMBER 2004
TO
IS 517 : 1986 SPECIFICATION FOR METHANOL
(METHYL ALCOHOL)**

(Second Revision)

[*Page 5, Table 1, Sl No. (iv), col 3*] — Substitute '0.03' for '0.0003'.

(*Page 5, Table 1*) — Insert the following after Sl No. (vii):

'(1)	(2)	(3)	(4)	(5)	(6)
viii)	Purity, percent by mass, <i>Min</i>	99.85	-	-	G'

(*Page 25, clause F-6.2*) — Insert the following Annex at the end of the text:

ANNEX G

[*Table 1, Sl No. (viii)*]

DETERMINATION OF ASSAY

G-1 GENERAL

This test method is related to determination of the purity of methyl alcohol by gas chromatography and, in addition, provides a means for identification of certain known impurities. Water and acidity are determined by other methods and the results are used to normalize the chromatographic values.

G-2 APPARATUS

G-2.1 Gas Chromatograph — Any gas liquid chromatograph having a flame ionization detector can be used with following accessories and operating conditions:

CONDITIONS:

Column	:	10 %, Carbowax 20
Mesh size	:	80/100
Column length	:	2 m
I.D.	:	3 mm

Amend No.1 to IS 517 : 1986

Column tubing Material	:	Stainless steel (tubing must be non-reactive with substrate, sample and carrier gas)
Carrier gas	:	Nitrogen (minimum purity – 99.95 mole %)
Syringe	:	10 μ
Sample size	:	0.2 μ
Electronic Integrator	:	For computation of results

CONDITIONS:

Column	:	10 %, Carbowax 20
Mesh size	:	80/100
Column length	:	2 m
I.D.	:	3 mm
Column tubing Material	:	Stainless steel (tubing must be non-reactive with substrate, sample and carrier gas)

NOTE — The above gas chromatographic conditions are suggestive. However, any GC with different columns may be used provided standardization/calibrations are done after setting up chromatographic conditions for the required resolution.

G-3 IDENTIFICATION, CALIBRATION AND STANDARDIZATION

Install the column in the chromatograph. Set the conditions given above of column temperature and carrier gas flow that gives the necessary resolution of the components in the sample being analyzed. Set the conditions as such which gives a 10 percent, *Min* recorder deflection for a 0.1 percent concentration of purity at the most sensitive setting of instrument. Allow sufficient time for the instrument to reach equilibrium as indicated by stable base line. Adjust carrier gas flow rate to a constant value.

G-3.1 Identification

Determine the retention time of each component by injecting small amount either separately or in known mixture.

G-3.2 Calibration and Standardization

G-3.2.1 The response of any given detector varies from one chemical to another. Therefore, calibration is must. Calibration allows the computation of response factor, which expresses the relative response of different components to the detector.

G-3.2.2 Calibration standard is prepared containing the impurities present in methanol. During standard preparation the exact amounts of all the components is noted. Inject 1.0 μ of calibration standard in the chromatographic column in accordance with the specified conditions.

G-3.2.3 Calculate response factor to four decimal places for all components relative to METHANOL using following equation:

$$RF_x = \frac{W_x \times A_m}{A_x \times W_m}$$

where

RF_x = response factor for component x relative to methanol;

W_x = weight of component x , in the calibration standard;

A_x = area of component x , in the calibration run;

W_m = weight of methanol in the calibration standard; and

A_m = area of methanol in the calibration standard run which is selected as reference component.

The component which is unknown in the sample is assigned the relative response factor 1.000 0. It is advisable that the determination of response factors be made on the basis of duplicate analysis.

G-3.2.4 Response factor should be re-checked after any perceptible change in column or instrument's performance.

G-4 PROCEDURE

Inject 1.0 μ l of sample into the chromatographic column using the same conditions as for components identification and standardization record and integrate the chromatogram using appropriate attenuation setting that provide optimum peak heights. Measure the area of all the peaks.

Amend No. 1 to IS 517 : 1986

G-5 CALCULATION

Calculate the concentrations of sample components using the following equation:

$$C_x, \text{ percent} = \frac{F_x \times R_x}{\sum R_x \times F_x} \times (100 - C)$$

where,

C_x = compound X , weight, percent;

R_x = area of compound X ;

F_x = relative response factor of compound X ;

$\sum R_x, F_x$ = sum of the individual component peak area R_x multiplied by their relative response factors F_x ; and

C = sum of acidity and water content of the sample, percent by mass.

G-6 PRECISION AND BIAS

G-6.1 Repeatability

Two results should be considered suspect if they differ by more than 0.000 6.

G-6.2 Reproducibility

Two results should be considered suspect if they differ by more than 0.002 7.

(PCD 10)

Indian Standard

SPECIFICATION FOR METHANOL (METHYL ALCOHOL)

(*Second Revision*)

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Indian Standard
SPECIFICATION FOR
METHANOL (METHYL ALCOHOL)
(Second Revision)

0. F O R E W O R D

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 30 June 1986, after the draft finalized by the Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first issued in 1954 stipulating the requirements and methods of test for three grades of methanol, namely, Grade 1, Grade 2 and Special. Through experience gained in the use of this standard and taking cognizance of the growth of additional demand and variety in the uses of methanol, it was revised in 1967. In the first revision, two grades, namely, methanol pure and technical were covered; the requirements of the special grade, mainly intended for Defence purposes were suitably covered under those of methanol, pure. High purity methanol required for the manufacture of antibiotics was also taken care of by prescribing additional requirements. Improvements in the test for alkalinity, a simpler alternative method for determination of sulphur and sulphur compounds and a revised scheme of sampling and criteria for conformity, were some of the salient features of this revision. The growing usage of methanol as a solvent in organic reactions has been safeguarded by prescribing Karl Fischer test for water content, wherever agreed. In drawing up this revision, the basic consideration was to stipulate the requirements on the basis of authentic data on the indigenously produced material and to incorporate all other changes found necessary as a result of experience gained through the use of the original standard. In order to take care of the present day requirements of some of the important users of methanol, this revision has been undertaken. In this revision, the requirements for specific gravity and distillation range have been modified accordingly.

0.3 To ensure co-ordination between standards prevailing in different countries, relevant overseas standards including documents of technical committee ISO/TC 47 Chemistry of the International Organization for Standardization, have also been consulted. This revised standard, it is

hoped, will assist the indigenous producers in controlling the quality of their production and consumers in selecting a material of acceptable quality intended for the specific end use envisaged.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for methanol (methyl alcohol).

2. GRADE

2.1 There shall be two grades of the material, namely :

- a) Methanol, pure; and
- b) Methanol, technical.

3. REQUIREMENTS

3.1 Description — The material shall be a clear and colourless liquid, free from matter in suspension.

3.2 Miscibility with Water — The material shall show no turbidity when one volume is mixed with 19 volumes of water at 27 ± 2 °C and examined as prescribed in Appendix A.

3.3 The material shall also comply with the requirements given in Table 1.

3.4 Methanol for Manufacture of Antibiotics — It shall, in general, comply with the requirements for methanol, pure, given in Table 1, except for the characteristics given in Table 2, for which separate requirements have been prescribed.

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in containers as agreed between the purchaser and the supplier, subject to the provisions of the law in force in the country for the time being.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR METHANOL

(Clause 3.3)

SL No.	CHARACTERISTIC	REQUIREMENT FOR GRADE		METHOD OF TEST, REF TO CL No. IN	
		Methanol, Pure	Methanol, Technical	Various Indian Standards	Appendix
(1)	(2)	(3)	(4)	(5)	(6)
i)	Relative density at 27°/27°C, <i>Max</i>	0.789	0.798	6 of IS : 82-1973*	—
ii)	Distillation range at 760 mm pressure	The difference between initial boiling point (IBP) and dry point (DP) shall not exceed 1°C including 64.5°C	Not less than 95 percent by volume shall distil between 65.5°C and 66.5°C [temperature being corrected for a pressure of 760 mm of Hg] (see Note below)	Method A of [P:18] of IS: 1448†	
iii)	Residue on evaporation, percent by mass, <i>Max</i>	0.01	0.015	8 of IS: 82-1973*	—
iv)	Alkalinity (as NH_3), percent by mass, <i>Max</i>	0.000 3	0.000 3	—	B-1
v)	Acidity, as acetic acid (CH_3COOH), percent by mass, <i>Max</i>	0.004	0.006	—	B-2
vi)	Aldehydes and ketones, as acetone (CH_3COCH_3), percent by mass, <i>Max</i>	0.03	0.100	—	C-1
vii)	Sulphur and compounds of sulphur (as S), percent by mass, <i>Max</i>	0.001	—	—	D

*Methods of sampling and test for thinners and solvents for paints (*first revision*).†Methods of test for petroleum and its products [P: 18] Distillation (*first revision*).

(Continued)

TABLE 1 REQUIREMENTS FOR METHANOL — *Contd*

Sl. No.	CHARACTERISTIC	REQUIREMENT FOR GRADE		METHOD OF TEST, REF TO CL NO. IN	
		Methanol, Pure	Methanol, Technical	Various Indian Standards	Appendix
(1)	(2)	(3)	(4)	(5)	(6)
viii)	Chlorine and chlorine compounds	To pass test	—	—	E
ix)	Water Content, percent by mass, <i>Max</i>	0.1	0.5	IS : 2362-1973*	—

NOTE.— In case the barometric pressure is lower than 760 mm Hg, use the correction to the observed temperature by adding 0.034°C for every mm below 760 mm. This correction is valid for a pressure range of 700 to 760 mm of Hg.

*Determination of water by the Karl Fischer method (first revision).

TABLE 2 SPECIAL REQUIREMENTS FOR METHANOL USED FOR ANTIBIOTICS
(Clause 3.4)

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN	
			IS : 82-1950*	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Acidity, as acetic acid (CH_3COCH), percent by mass, <i>Max</i>	0.002	—	B-2
ii)	Aldehydes and ketones as acetone (CH_3COCH_3), percent by mass, <i>Max</i>	0.001	—	C-2
iii)	Residue on evaporation, percent by mass, <i>Max</i>	0.001	8	—

*Methods of sampling and test for thinners and solvents for paints (first revision).

4.1.2 All containers, in which the material is packed, shall be dry, clean and free from substances soluble in methanol and shall be leak proof.

4.1.3 Necessary safeguards against the risks arising from the storage and handling of large volumes of flammable liquids shall be provided and

all precautions shall be taken at all times to prevent accident by fire or explosion.

4.1.4 Except when they are opened for the purposes of cleaning and rendering them free from vapour, all containers shall be kept securely closed, unless they have been thoroughly cleaned and freed from methanol vapour.

4.2 Marking

4.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

4.2.2 Each container shall be marked with the :

- a) Name of the material;
- b) Manufacturer's name or trade-mark, if any, or both;
- c) Gross, net and tare mass;
- d) Month and year of manufacture; and
- e) Minimum cautionary notice worded as under :
**'FLAMMABLE ! INGESTION OR ABSORPTION DANGEROUS !
 CAUSES BLINDNESS ! CUMULATIVE POISON !'**

4.2.3 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Marks on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or processors may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in Appendix F.

6. TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed in appendices and various Indian Standards. References to various standards and relevant appendices are given in col 5 and 6 of Table 1, and col 4 and 5 of Table 2.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

APPENDIX A

(*Clause 3.2*)

TEST FOR MISCIBILITY WITH WATER

A-1. PROCEDURE

A-1.1 Mix 10 ml of the material with 190 ml of distilled water in a suitable glass vessel and allow to stand at any temperature within the range $27 \pm 2^{\circ}\text{C}$ for one hour. Compare the clarity of the mixture with that of an equal volume of distilled water.

A-1.1.1 The material shall be taken to comply with the requirement if there is no noticeable difference in clarity between the mixture and water.

APPENDIX B

[*Table 1, Items (iv) and (v) and Table 2, Item (i)*]

DETERMINATION OF ALKALINITY AND ACIDITY

B-1. ALKALINITY

B-1.1 Reagents

B-1.1.1 Methyl Red Solution — 0.15 g in 500 ml of water (*see* IS : 2263-1962†).

B-1.1.2 Standard Sulphuric Acid — 0.01 N.

B-1.2 Procedure — Place 100 ml of water and a few pieces of clean porous porcelain in a 500-ml resistance glass conical flask and boil gently for 5 minutes to eliminate carbon dioxide. Cool with the neck of the flask closed with a stopper carrying a soda lime tube. Add to it one drop of methyl red solution and titrate to a faint pink colour with standard

*Specification for water for general laboratory use (*second revision*).

†Methods of preparation of indicator solutions (*first revision*).

sulphuric acid. To this, add 100 ml of the material and titrate to the same faint pink colour with standard sulphuric acid. Calculate the alkalinity.

B-1.3 Calculation

$$\text{Alkalinity (as NH}_3\text{), percent by mass} = \frac{1.7 V N}{100 S}$$

where

V = volume in ml, of standard sulphuric acid solution used in titration with material;

N = normality of standard sulphuric acid solution; and

S = specific gravity of the material at the temperature of determination.

B-2. ACIDITY

B-2.1 Reagents

B-2.1.1 Phenolphthalein Indicator — prepared by dissolving 0.1 g of phenolphthalein in 100 ml of 60 percent ethyl alcohol (see IS : 2263-1979*).

B-2.1.2 Standard Sodium Hydroxide Solution — 0.01 N.

B-2.2 Procedure — Place 100 ml of carbon dioxide free water as prepared in B-1.2 in a 500-ml resistance glass conical flask. Cool slightly and add 100 ml of the material. Boil gently for a further period of 5 minutes. At the end of this period, close the neck of the flask with a stopper carrying a soda-lime tube and allow to cool. When cold, remove the stopper, add 0.5 ml of phenolphthalein indicator and examine for alkalinity; if not alkaline, titrate with standard sodium hydroxide solution using a micro-burette.

B-2.3 Calculation

$$\text{Acidity (as CH}_3\text{COOH), percent by mass} = \frac{6 V N}{100 S}$$

where

V = volume in ml, of standard sodium hydroxide solution used;

N = normality of standard sodium hydroxide solution; and

S = specific gravity of the material at the temperature of determination.

*Methods of preparation of indicator solutions (first revision).

APPENDIX C

[Table 1, Item (vi) and Table 2, Item (ii)]

DETERMINATION OF ALDEHYDES AND KETONES

C-1. HYDROXYAMMONIUM CHLORIDE METHOD

C-1.0 Outline of the Method — Reaction with hydroxylamine hydrochloride forms the basis for determination of carbonyl function (aldehydes and ketones). Condensation products called oximes are produced. The hydrochloric acid released in the reaction is titrated with standard sodium hydroxide solution using bromophenol blue as indicator.

C-1.1 Reagents

C-1.1.1 Standard Sodium Hydroxide Solution — 0.1 N.

C-1.1.2 Rectified Spirit — 95 percent by volume (see IS : 323-1959*).

C-1.1.3 Bromophenol Blue Indicator — Dissolve 0.2 g of bromophenol blue in 3 ml of the standard sodium hydroxide solution and dilute to 100 ml with rectified spirit.

C-1.1.4 Hydroxylamine Hydrochloride Solution — Dissolve 4 g of hydroxylamine hydrochloride in 20 ml of water. Dilute this to 200 ml with rectified spirit, heat on a boiling water-bath for 30 minutes. Cool and add 5 ml of the bromophenol blue indicator with just sufficient sodium hydroxide solution (2 N) to impart a dichroic yellow-green colour to the liquid.

C-1.2 Procedure — Measure 25 ml of the material into a 150 ml conical flask, add 25 ml of hydroxylamine hydrochloride solution and, after loosely stoppering, heat on a boiling water-bath for 10 minutes. Cool and titrate with standard sodium hydroxide solution until as near a match as possible is obtained with a control made by mixing 25 ml of distilled water with 25 ml of hydroxylamine hydrochloride in a similar 150-ml conical flask.

C-1.3 Calculation

$$\begin{array}{l} \text{Aldehydes and ketones (as acetone) ,} \\ \text{percent by mass} \end{array} = \frac{0.023 (V - 0.6)}{S}$$

where

V = volume in ml of standard sodium hydroxide solution, and

*Specification for rectified spirit (revised).

S = specific gravity of the material taken at the temperature of the test.

NOTE — The correction 0.6 is subtracted from the number of millilitres of sodium hydroxide solution used, to allow for the effect on the indicator of different alcohol concentration in the experiment and control.

C-2. DINITROPHENYLHYDRAZINE METHOD

C-2.0 Outline of the Method — In acid medium, the carbonyl compounds present are converted with dinitrophenylhydrazine into the corresponding 2, 4-dinitrophenyl hydrazones. In alkaline medium, these have a red colour, which is compared visually with colours produced by standard solution; or alternatively, the optical density is measured and the content of carbonyl compounds is estimated by reference to a standard curve.

C-2.1 Apparatus

C-2.1.1 Water-Bath — controlled at 60 °C.

C-2.1.2 Graduated Flask — 25-ml capacity.

C-2.1.3 Photoelectric Absorptiometer or Spectrophotometer — with 0.5 cm cells. Alternatively, flat bottom tubes of 20-ml capacity shall be used.

C-2.2 Reagents

C-2.2.1 Carbonyl-Free Methanol — Reflux 1 000 ml of methanol with 5 g of 2,4-dinitrophenylhydrazine and 5 drops of concentrated hydrochloric acid (sp gr 1.18) for 2 to 3 hours. Distil off methanol. Reject the first 100 ml and collect the next 800 ml, rejecting the remainder. If, in spite of the precautions taken, the distillate is found to be coloured, then it may be redistilled.

C-2.2.2 Concentrated Hydrochloric Acid — specific gravity 1.18 (see IS : 265-1976*).

C-2.2.3 Dinitrophenylhydrazine Solution — Dissolve 0.03 g of 2,4-dinitrophenylhydrazine in 40 ml of the carbonyl-free methanol containing 0.3 ml of concentrated hydrochloric acid (specific gravity 1.18) and dilute to the mark in a 50 ml one-mark volumetric flask with the carbonyl-free methanol. Prepare this solution fresh each day.

C-2.2.4 Potassium Hydroxide Solution, Freshly Prepared — Dissolve 10 g of potassium hydroxide in 10 ml of water, cool and dilute to the mark in a 50-ml one-mark volumetric flask with the carbonyl-free methanol.

*Specification for hydrochloric acid (second revision).

C-2.2.5 Standard Acetone Solution — Weigh 1.000 g of acetone and dilute to the mark in a one-mark 100-ml volumetric flask with carbonyl-free methanol. Dilute 1.0 ml of this solution to 100 ml with the carbonyl-free methanol. One ml of the diluted solution contains 0.1 mg of acetone.

C-2.3 Procedure

C-2.3.1 Instrumental Procedure — Prepare five solutions by diluting 1.0, 2.0, 4.0, 8.0 and 10.0 ml portions of the standard acetone solution to 25.0 ml with the carbonyl-free methanol. To 1.0 ml of each of the solutions thus obtained, contained in a test tube fitted with a ground glass stopper, add 1.0 ml of the 2,4-dinitrophenylhydrazine solution. Stopper the tube and heat for 50 minutes in the water-bath at 60°C, cool, add 8.0 ml of the potassium hydroxide solution and after 5 to 15 minutes measure the optical density of each solution at a wavelength of 430 m μ using as a blank 1.0 ml of the carbonyl-free methanol treated in the same way. Prepare a calibration chart by plotting weights (in milligrams) of acetone against corresponding values of optical density.

Dilute 5.0 ml of the sample to 25.0 ml with the carbonyl-free methanol. Transfer 1.0 ml of this solution to a test tube fitted with a ground glass stopper and add 1.0 ml of the 2,4-dinitrophenylhydrazine solution. Stopper the tube and heat for 50 minutes in the water-bath at 60°C; cool, add 8.0 ml of the potassium hydroxide solution and after 5 to 15 minutes, measure the optical density of the solution at a wavelength of 430 m μ using as a blank 1.0 ml of the carbonyl-free methanol treated in the same way. By reference to the calibration chart prepared as described above, read the acetone content (in milligrams) of the solution.

C-2.3.2 Visual Procedure — Dilute 5.0 ml of the sample to 25.0 ml with the carbonyl-free methanol and transfer 1.0 ml of this solution to a test tube fitted with a ground glass stopper. At the same time, prepare five solutions by diluting 1.0, 2.0, 4.0, 8.0 and 10.0 ml portions of the standard acetone solution to 25.0 ml with the carbonyl-free methanol. Transfer 1.0 ml of each of the solutions so obtained to a series of test tubes fitted with ground glass stoppers and treat each of the solutions in the following manner.

Add 1 ml of 2,4-dinitrophenylhydrazine solution. Stopper the tube and heat for 50 minutes in the water-bath at 60°C; cool and add 8.0 ml of potassium hydroxide solution.

After 5 to 15 minutes, compare the colour of the solution containing the sample with the series of prepared colour standards in 20 ml flat bottom tubes, noting the acetone content of the standard that most closely matches the sample solution. In the event of doubt as to which of two

colours standard most closely matches the sample solution, take the mean of the two acetone contents.

C-2.4 Calculation

C-2.4.1 Instrumental Procedure — Carbonyl compounds, calculated as acetone :

$$\begin{array}{l} \text{Carbonyl compounds (as CH}_3\text{COCH}_3 \text{),} \\ \text{percent by mass} \end{array} = \frac{0.5 \times M}{S}$$

where

M = mass in milligrams of acetone corresponding to the optical density recorded; and

S = specific gravity of the sample.

C-2.4.2 Visual Procedure — Carbonyl compounds, calculated as acetone:

$$\begin{array}{l} \text{Carbonyl compounds (as CH}_3\text{COCH}_3 \text{),} \\ \text{percent by mass} \end{array} = \frac{0.002 \times V}{S}$$

where

V = volume in ml of the standard acetone solution used in preparing the colour standard that most closely matches the sample, and

S = specific gravity of the sample.

APPENDIX D

[Table 1, Item (vii)]

DETERMINATION OF SULPHUR AND COMPOUNDS OF SULPHUR

D-0. General

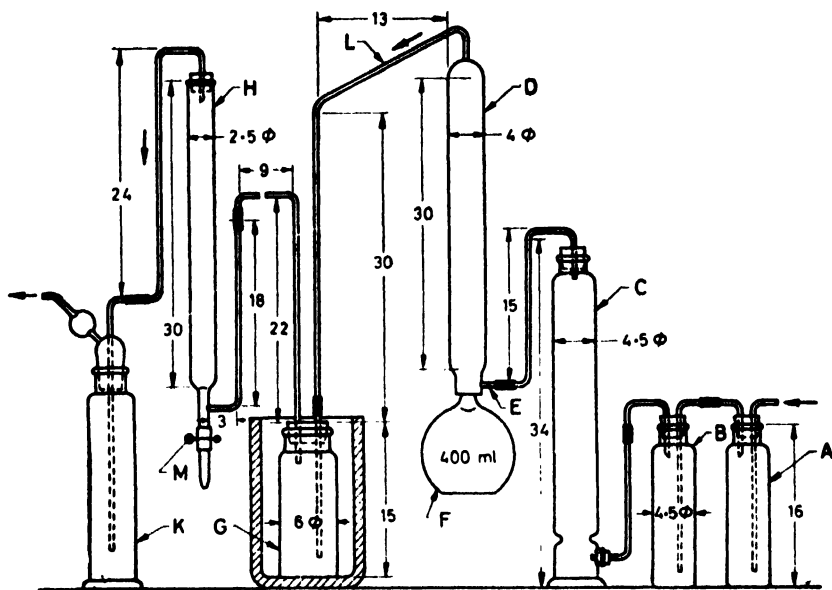
D-0.1 Two alternative methods (D-1 and D-2) for determination of sulphur and compounds of sulphur have been prescribed in this appendix. The choice between these, shall be subject to agreement, except that while stating the requirements of the material against this characteristic; the method of determination used shall be clearly stated.

D-1. COMBUSTION METHOD

D-1.0 Outline of the Method — A known quantity of the material is burnt in a lamp; the products of combustion being drawn through sodium peroxide solution and the resulting sulphate being determined by nephelometric method.

D-1.1 Apparatus

D-1.1.1 Air Purifying Train — It shall consist of two bottles *A* and *B* and a tower *C*, of the dimensions shown in Fig. 1, all packed with pumice. The pumice in *A* and *C* shall be soaked with potassium hydroxide solution, so that its depth in the former is 2 to 3 cm, and in the latter just sufficient to cover the inlet tube. The pumice in *B* shall be soaked with lead acetate solution to a depth of 2 to 3 cm. The inlet tubes in *A* and *B* shall reach within a few millimetres of the bottom.



All dimensions in Centimetres

FIG. 1 AIR PURIFYING TRAIN

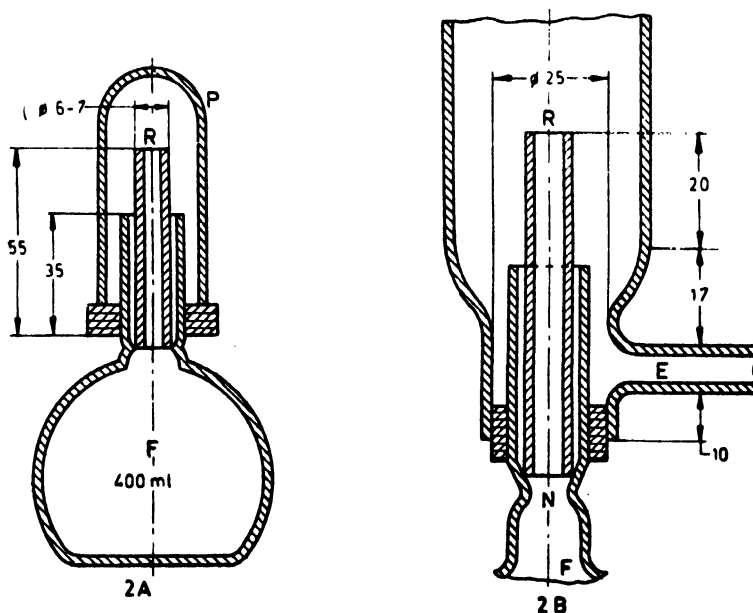
This arrangement removes acidic sulphur compounds only and if the air contains such compounds as carbon disulphide or thiophene, the train shall be preceded by a length of combustion tubing, packed, with coarse copper oxide, and heated to dull redness. In that case, the bottle *B* shall contain potassium hydroxide solution instead of lead acetate solution.

D-1.1.2 Chimney and Absorption Train — The air from the tower *C* shall be introduced by the side-tube *E* to the foot of the chimney *D*, into which the lamp *F* fits with a rubber stopper. The products of combustion

shall be drawn up the chimney *D* and from there into the first absorption bottle *G*, which shall consist of a 500-ml wide-mouthed bottle, immersed in an ice-bath. The connection shall be sloped at *L* to avoid the possibility of condensed water running down the hot chimney. The ice-bath condenses the water and also cools the gases before they enter the sodium peroxide solution, thus avoiding undue decomposition of the latter.

From the absorption bottle *G*, the gases shall pass through the tower *H*, containing short pieces of glass rod. The train shall be completed with the Drechsel wash-bottle *K* of about 250-ml capacity. The bottles *G* and *K*, and the tower *H* shall each contain 25 ml of sodium peroxide solution. The liquid from *H* can be drained by pinch-cock *M*.

D-1.1.3 Lamp — The lamp *F* as shown in Fig. 2A shall consist of a round flask of about 400-ml capacity. The neck of the flask shall be constricted at *N* to support the tube *R* in which shall be loosely fitted an ordinary cylindrical cotton-wick. The length of *F* shall be such that when the lamp is in position, the top *R* shall be about 4 cm above the top of *E* (see Fig. 2B). During weighing, etc, a test tube *P* shall be inverted over the wick as a sheath (see Fig. 2A).



All dimensions in millimetres.

FIG. 2 LAMP

D-1.1.4 Nessler Tubes — Two tubes of thin transparent glass, about 25 mm in diameter and about 150 mm in length, graduated up to 50 ml. The depth, measured internally from the graduated mark to the bottom, shall not vary by more than 2 mm in the tubes used.

D-1.2 Regents

D-1.2.1 Absolute Alcohol — conforming to IS : 321-1964*.

D-1.2.2 Potassium Hydroxide Solution — 50 percent (*m/v*).

D-1.2.3 Lead Acetate Solution — 10 percent (*m/v*).

D-1.2.4 Sodium Peroxide Solution — Dissolve cautiously 10 g of sodium peroxide in 500 ml of ice cold water.

D-1.2.5 Concentrated Hydrochloric Acid — specific gravity 1.16 (conforming to IS : 265 - 1976†).

D-1.2.6 Barium Chloride Solution — 12 percent (*m/v*) of barium chloride crystals dissolved in water.

D-1.2.7 Standard Sulphate Solution — Acidify 75 ml of sodium peroxide solution with concentrated hydrochloric acid, bring to boil and then cool. Transfer the solution to a 500-ml volumetric flask. Add 15.6 ml of 0.01 N standard sulphuric acid and make up the volume to 500 ml mark.

D-1.3 Procedure

D-1.3.1 Cleaning of Wick — Boil the wick for an hour with soap solution to remove oily matter and size (which might contain sulphur compounds), then boil it for several minutes with successive changes of distilled water until the water remains clear and bright. Squeeze out the wick, wash it twice in absolute alcohol, dry at $100 \pm 2^{\circ}\text{C}$ and insert in tube *R*.

D-1.3.2 Transfer 250 g of the material to the lamp *F*, pouring a little down the tube *R*, replace the sheath and weigh. Attach the wash-bottle *K* to a suction pump, light the lamp, draw a rapid stream of air through the apparatus and quickly push the lamp into position. The initial stream of air shall not be too slow as otherwise the lamp may be extinguished. Once the test is under way, reduce the air stream a little but take care that it is rapid enough to keep the lamp burning vigorously. When all the sample has been burned, which will take about 24 hours, stop the flow of air through the apparatus, remove the lamp, replace the sheath and weigh. The difference between the two weighings, *M*, is the mass of material consumed.

*Specification for absolute alcohol (revised).

†Specification for hydrochloric acid (second revision).

D-1.3.3 Transfer the contents of the vessels *G*, *H* and *K* to a beaker. If necessary, boil until the volume of the liquid is reduced to about 450 ml, then add 10 ml of concentrated hydrochloric acid. Filter and again bring it to boil. Cool the solution and make up the volume to 500 ml in a volumetric flask.

D-1.3.4 Pipette out 50 ml of the test solution (**D-1.3.3**) into a Nessler tube and add 2 ml of barium chloride solution. Carry out a control test in another Nessler tube using 50 ml of standard sulphate solution if *M* is equal to 250 g (or a proportionate lower or higher volume) and 2 ml of barium chloride solution. Mix both the solutions well, stir and compare the turbidity produced, if any, after five minutes.

D-1.3.5 The material shall be considered to have satisfied the specified requirement if the turbidity produced in the test with the material is not greater than that produced in the control test.

D-2. REDUCTION METHOD

D-2.0 Outline of the Method — The sulphur present in the material is reduced with Raney nickel to metal sulphide, which is heated with hydrochloric acid, the hydrogen sulphide evolved is absorbed in sodium hydroxide solution and titrated with standardized mercuric acetate solution in the presence of dithizone as indicator. This method determines all sulphur compounds normally encountered in methyl alcohol for industrial uses. It does not, however, determine alkyl sulphur and other oxygen bounded sulphur compounds.

D-2.1 Apparatus

D-2.1.1 Reduction Apparatus — see Fig. 3.

D-2.1.2 Burette — Class A, 10-ml capacity, conforming to IS : 1997-1982* and graduated in 0.05 ml.

D-2.2 Reagents

D-2.2.1 Nitrogen Gas — free from carbon dioxide.

D-2.2.2 Acetone — conforming to IS : 170-1986†.

D-2.2.3 Raney Nickel — Treat 10 g of 50/50 nickel/aluminium alloy with 100 ml of 10 percent (*m/v*) sodium hydroxide solution, cooling with ice while the reaction proceeds. Wash the Raney nickel three times with water, remove the water by decantation and store the Raney nickel under

*Specification for burettes (*second revision*).

†Specification for acetone (*third revision*).

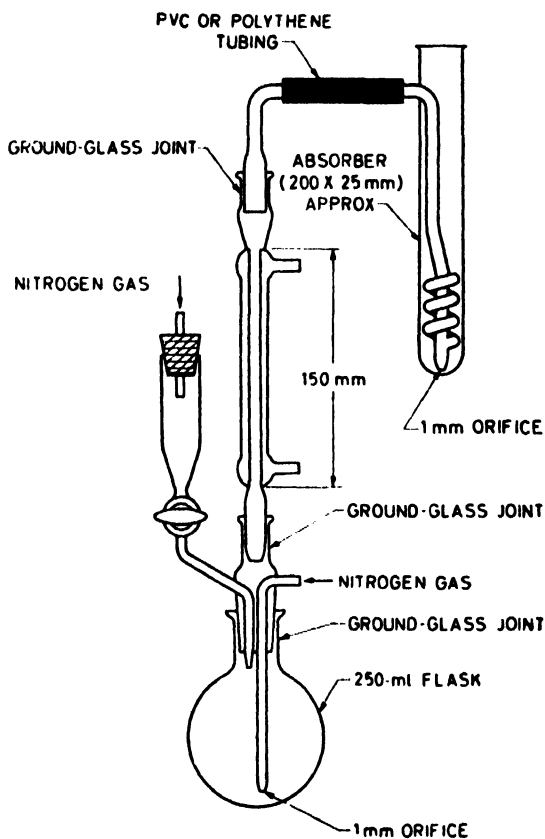


FIG. 3 REDUCTION APPARATUS FOR USE IN THE DETERMINATION OF SULPHUR AND SULPHUR COMPOUNDS

100 ml of isopropyl alcohol. Raney nickel prepared in this way retains its activity for about three days.

5 ml of this suspension will reduce quantitatively 1.0 to 1.5 mg of organic sulphur to hydrogen sulphide.

A stabilized form of Raney nickel is available commercially, and may be prepared by warming half pellet (0.24 g) in the reaction flask, in 10 ml of acetone. When the pellet has disintegrated, decant the acetone and introduce the sample.

NOTE — Raney nickel is spontaneously flammable in air.

D-2.2.4 Sodium Hydroxide Solution — 1.0 N.

D-2.2.5 Hydrochloric Acid — (sp gr 1.18) diluted (3 : 2) with water.

D-2.2.6 Mercuric Acetate Solution — Dissolve 0.200 g of mercuric oxide in 25 ml of water containing 1 ml glacial acetic acid, and dilute to 1 000 ml with water and standardize as follows.

D-2.2.6.1 Standardization of the mercuric acetate solution — Add a known amount of an aliphatic sulphur compound (di-isoamyl sulphide) to the sulphur-free methanol. Carry out a determination by the method (described in D-2.3) using an aliquot of this solution representing 130 to 170 μ g of sulphur. From the volume of the mercuric acetate solution required in the titration, calculate the mass, in micrograms, of sulphur equivalent to 1 ml of the mercuric acetate solution.

D-2.2.7 Methanol, Sulphur-Free — Into a 2-litre, two-necked round bottom flask, measure 20 ml of acetone and add 0.5 g (1 pellet) of Raney nickel or 10 ml of the suspension. Warm until the pellet has disintegrated and decant the acetone. Add 1 500 ml of methanol and reflux on a water-bath. Insert the nitrogen lead in the second neck and pass in gas at the rate of 2 bubbles per second.

Heat until slight effervescence is observed on the methanol surface and continue for 30 minutes. Turn off the gas flow and the heating, and allow the nickel to settle. Decant the methanol into a flask and distil. Reject the first 250 ml and collect the next 1 000 ml, rejecting the remainder.

D-2.2.8 Dithizone — One percent (m/v) solution in acetone.

D-2.3 Procedure — Place 5 ml of Raney nickel suspension, or half pellet as described in the flask, then add 100 ml of the sample. Place the flask on a water-bath and pass nitrogen through at the rate of about 3 litres per hour. Adjust the temperature of the heating mantle until slight effervescence appears on the surface (just below boiling point). Continue for 50 minutes, swirling the flask at intervals.

Place 10 ml of the sodium hydroxide solution and 10 ml of acetone in the absorber.

Place 15 ml of the hydrochloric acid in the dropping funnel, switch off the heating mantle, and attach a second nitrogen lead to the glass tube in the top of the dropping funnel. Admit the acid, a few millilitres at a time, to the flask. Add two drops of the dithizone solution and one drop of the mercuric acetate solution to the absorber using the burette. The colour change should be from yellow to pink. Liberation of hydrogen sulphide reverses the colour change; when all the acid has been added, increase the nitrogen flow rate to about 12 litres per hour, switch on the

heating mantle again, continue heating for a further 20 minutes, titrating with the mercuric acetate solution as necessary. When all the hydrogen sulphide has apparently been titrated, disconnect the flow of nitrogen momentarily, cool the flask by blowing air on it and allow the absorbent to travel up the delivery tube in the absorber, to recover any hydrogen sulphide which may be absorbed on its surface.

Carry out a blank test on the Raney nickel and the sulphur-free methanol.

D-2.4 Calculation — Sulphur and sulphur compounds are calculated as :

$$S, \text{ parts per million by mass} = \frac{M \times (V_2 - V_3)}{100 \times s}$$

where

M = mass in micrograms, of sulphur equivalent to 1 ml of the mercuric acetate solution (determined as in D-2.2.6.1);

V_2 = volume, in ml, of the mercuric acetate solution required in the test determination;

V_3 = volume, in ml, of the mercuric acetate solution required in the blank test; and

s = specific gravity of the sample.

APPENDIX E

[Table 1, Item (viii)]

DETERMINATION OF FREEDOM FROM CHLORIDE AND CHLORINE COMPOUNDS

E-1. REAGENTS

E-1.1 Alcoholic Caustic Potash Solution — 0.1 N, approximately.

E-1.2 Dilute Sulphuric Acid — 5 N, approximately.

E-1.3 Litmus Paper — blue.

E-1.4 Sulphur Dioxide Water — saturated solution.

E-1.5 Concentrated Nitric Acid — sp gr 1.42 (see IS : 264-1976*).

E-1.6 Silver Nitrate Solution — 4 percent (m/v).

*Specification for nitric acid (second revision).

E-2. PROCEDURE

E-2.1 Reflux 50 ml of the material with 10 ml of alcoholic caustic potash solution for 1 hour in a 250-ml conical flask fitted with an upright condenser. Transfer the contents to a 250-ml beaker and wash down the flask and the condenser with 50 ml water into the beaker. Drive off the alcohol on a water-bath and evaporate the solution down to about 20 ml. Cool the beaker and acidify the solution faintly with dilute sulphuric acid using litmus paper as indicator. Add a drop of sulphur dioxide water and mix the contents thoroughly. Add 1 ml of concentrated nitric acid and boil to drive off nitrous fumes, if any. Cool the solution and filter, if necessary. To the clear filtrate, add 5 ml of silver nitrate solution.

E-2.2 Carry out a blank test substituting the material by the same quantity of distilled water.

E-2.3 The material shall be considered as having satisfied the requirements prescribed in Table 1, if any opalescence produced in the test with the material is not greater than that produced in the blank test.

APPENDIX F

(*Clause 5.1*)

SAMPLING OF METHANOL**F-1. GENERAL REQUIREMENTS OF SAMPLING**

F-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

F-1.1 Samples shall be taken in a protected area with good ventilation. The samples shall be kept away from flames.

F-1.2 The sampling instrument shall be clean and dry.

F-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

F-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring, or both or by rolling so as to bring all portions into uniform distribution.

F-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass containers.

F-1.6 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

F-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with the manufacturer's name or trade-mark, the month and year of manufacture of the material, the batch number (if available), and other details of sampling, such as the date of sampling and sampler's name.

F-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

F-2. SAMPLING INSTRUMENTS

F-2.0 The following forms of sampling instruments may be used :

- a) Weighted sampling can for taking samples from various depths in large tanks, and
- b) Sampling tube.

F-2.1 Weighted Sampling Can — of suitable capacity, 500 to 1 000-ml and of such a mass as to sink readily in the material to be sampled. It has a long chain or cord attached to permit filling at any desired level (*see* Fig. 4). The metal used to weigh the apparatus shall be fitted externally, as irregularities in the metal are likely to contaminate the sample, if the weight is fitted internally.

F-2.2 Sample Tube — It is made of metal or thick glass and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 5). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

F-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

F-3. SCALE OF SAMPLING

F-3.1 Lot — In any consignment, all the containers of the same size and grade belonging to the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

F-3.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot

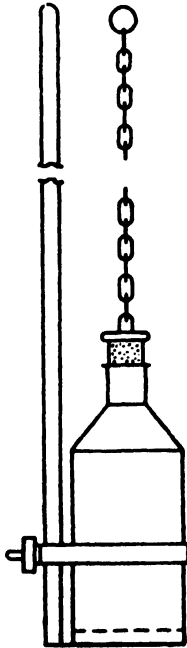
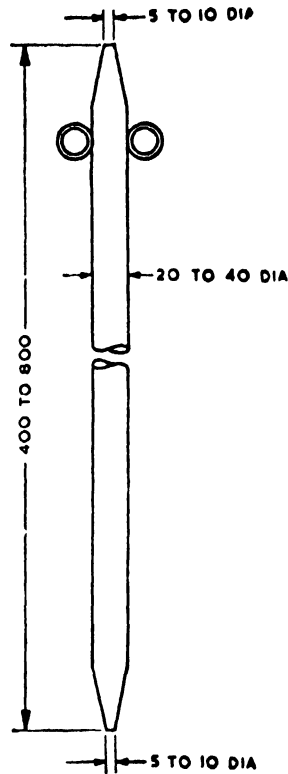


FIG. 4 WEIGHTED SAMPLING CAN



All dimensions in millimetres.

FIG. 5 SAMPLING TUBE

separately. For this purpose, the number of containers to be selected from a lot shall be in accordance with Table 3.

TABLE 3 SCALE OF SAMPLING

NO. OF CONTAINERS IN THE LOT N	NO. OF CONTAINERS TO BE SELECTED n
(1)	(2)
Up to 25	3
26 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

F-3.2.1 The containers shall be chosen at random from the lot. To ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed.

F-4. TEST SAMPLES AND REFEREE SAMPLES

F-4.1 Preparation of Sets of Samples for Testing Individually — Draw with an appropriate sampling instrument (*see F-2*), equal portions of the material from different parts of each container selected as in F-3.2 and freshly opened. Mix these small portions of the material from the same container to obtain a sample, representative of the container. Keep these representative samples from different containers separately. From each representative sample, draw three equal portions of the material, each sufficient for carrying out the intended tests and transfer them into thoroughly cleaned and dry sample containers. Seal the sample containers air-tight. Thus three sets of test samples are obtained such that each set has a test sample from each selected container. Send one each of these sets to the purchaser and the supplier. Reserve the third set as referee sample bearing the seals of the purchaser and the supplier. Keep the referee sample at a place agreed to between the purchaser and the supplier.

F-4.2 Preparation of Composite Samples — Draw, with an appropriate sampling instrument (*see F-2*), equal portions of material from each container selected as in F-3.2 and mix them together thoroughly to constitute a single composite sample. Divide this composite sample into three parts, each sufficient for carrying out the intended tests and transfer them to thoroughly cleaned and dry sample containers. Send one each of these to the purchaser and the supplier. Reserve the third composite sample as referee sample at a place agreed to between the purchaser and the supplier.

F-5. NUMBER OF TEST

F-5.1 Individual Samples — Test for distillation range, specific gravity, aldehydes and ketones (as acetone) shall be carried out individually on each of the test samples in a set prepared in F-4.1.

F-5.2 Composite Samples — Tests for the determination of the remaining characteristics of this specification shall be carried out on the composite sample prepared as in F-4.2.

F-6. CRITERIA FOR CONFORMITY

F-6.1 For Composite Samples — The lot shall be declared as conforming to the requirements of this specification if each of the test results made

*Methods for random sampling.

on the composite sample (F-5.2) satisfies the corresponding requirements of this specification.

F-6.2 For Individual Samples — The lot shall be declared as conforming to this specification if for each of the characteristics tested on the individual samples according to F-5.1, the mean and the range of test results be calculated as below:

Mean ($\bar{x}$) = sum of the test results divided by the number of test results so added; and

Range (R) = difference between the maximum and minimum values of the test results.

The expression ($\bar{x} - 0.6R$) and ($\bar{x} + 0.6R$) shall then be calculated. The lot shall be considered to conform in respect of the relevant characteristics, if ($\bar{x} + 0.6R$) is less than the maximum specified value and ($\bar{x} - 0.6R$) is greater than the minimum specified value. When the number of container selected is ten, it shall be divided into two groups of five each. Taking first five in one group and next five in other group, and for each group, range and its average shall be calculated. The mean range is:

$$\bar{R} = \frac{\text{Sum of the ranges of two groups}}{2}$$

In the expression $\bar{x} - 0.6R$, R shall be replaced by $\bar{R}$ to determine the conformity of the lot.

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(Continued from page 2)

Panel for Monohydric Alcohols, PCDC 10 : P1

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 323 0131, 323 3375, 323 9402

Fax : 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams : Manaksanstha
(Common to all Offices)

Central Laboratory :

Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010

Telephone

8-77 00 32

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 323 76 17

*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054 337 86 62

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022 60 38 43

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 235 23 15

†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East),
MUMBAI 400093 832 92 95

Branch Offices::

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001 550 13 48

‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road,
BANGALORE 560058 839 49 55

Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003 55 40 21

Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001 40 36 27

Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037 21 01 41

Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001 8-28 88 01

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 8-71 19 96

53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003 54 11 37

5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 20 10 83

E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001 37 29 25

117/418 B, Sarvodaya Nagar, KANPUR 208005 21 68 76

Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road,
LUCKNOW 226001 23 89 23

NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010 52 51 71

Patliputra Industrial Estate, PATNA 800013 26 23 05

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*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street,
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